

MICHOCOPY RESOLUTION TEST CHART



FINAL REPORT

on

CRYOCOOLER SYSTEMS DEMONSTRATION

Contract No. N00014-84-C-0397 TRW Project No. 44236

Period-of-Performance 4 September 1984 - 23 February, 1986

Submitted to

The Office of Naval Research 800 N. Quincy St. Arlington, VA 22217

Prepared by

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31 August, 1987



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SUMMARY

This is the Final Report on Contract No. N00014-84-C-0397 on the integration and evaluation of a small plastic Stirling-cycle cryocooler and SQUID gradiometer. TRW successfully transferred Stirling-cycle cryocooler technology from its development at NBS to industry. We installed, modified, and operated a small, plastic, five-stage Stirling cycle refrigerator which was developed at the National Bureau of Standards and furnished to TRW for this project. We also designed and fabricated a new gas handling system, pressure wave generator and displacer drive, and fiberglass-epoxy sleeves for another cryocooler. We successfully designed, fabricated, and operated a monolithic thin film SQUID gradiometer on a single silicon chip. The cooler was modified by the addition of a 4 kelvin Joule-Thompson stage before delivery to TRW and we initially fabricated this SQUID with a Nb-Pb technology for 4 kelvin experiments. We later transferred to an all-niobium technology developed at TRW when it was decided to operate the cryocooler at about 7 kelvin without the Joule-Thomson stage. We designed, constructed, and installed all components necessary to interface the magnetometer with the cryocooler. The silicon chip magnetometer was mounted on a silicon printed circuit board which was heat-sunk to the final stage of the cryocooler. Radiation shields were constructed and low noise and low thermal electrical leads were provided.

1. Introduction

The objectives of this program were to design, fabricate, and integrate a thin film SQUID gradiometer with a small plastic Stirling cryocooler and evaluate the performance. This required the transfer of small closed-cycle cryogenic refrigerator technology from the laboratory to industry and evaluate an operational cryocooler/magnetometer system. We designed and fabricated a monolithic thin film dc SQUID magnetometer, interfaced the magnetometer to the Stirling closed-cycle cryocooler built and furnished by the National Bureau of Standards (NBS). inductance of the two-junction, dc SQUID is split into two parallel loops and is insensitive to a uniform field applied perpendicular to the plane loops. However it responds to a difference in field applied to the two coils (dHz/dx). In order to increase the magnetic field gradient sensitivity, superconducting dc transformers were used to couple flux from large (9mm by 3mm) input pick-up loops to the small SQUID inductance. magnetometer was designed to fit on a one centimeter square silicon chip.

We successfully installed and operated the five stage Stirling-cycle helium refrigerator developed at NBS. The cryocoler uses two pneumatically driven diaphragm compressors which operate on the He gas. These diaphragm compressors, the pressure wave generator (PWG) and the displacer drive (DD), are controlled by solenoid valves which determine the timing and duration of their strokes. A Commodore 64 computer was chosen as an inexpensive, versatile controller to operate the cryocooler valves. Through a suitable interface the binary output of the computer operates the solenoid valves and thus the frequency of operation and the timing of the phases in the cycle. Timing could easily be changed in software by changing a byte in the memory.

The cryocooler was operated continuously for longer than three weeks on several occasions and achieved a minimum temperature below 7 kelvin. However, the operating temperature was neither reproducible nor extremely stable. The cryocooler required frequent fine adjustment of the manual needle valves to obtain optimum operation.

The cryocooler received from NBS had an additional helium gas Joule-Thompson stage in order to reach 4 K and be able to operate Pb-alloy based Josephson devices. Because of the difficulty in operating the miniature J-T valve successfully, we removed the J-T from the cryocooler and switched from the Nb-Pb alloy to an all-Nb process for fabricating the SQUID which could then operate at higher temperatures.

As a part of this program, we also designed and fabricated new components for the pressure wave generator and the displacer drive, and the fiberglass-epoxy sleeves for another cryocooler assembly. These components were designed in consultation with Dr. J. E. Zimmerman at NBS, who designed and built the first

cryocooler. We also designed and constructed an entirely new compressor/gas handling system.



2. SQUID Magnetometer

2.1 SQUID Design

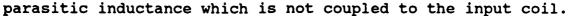
Figure 1 is a schematic diagram of the planar thin film gradient dc SQUID which we designed and constructed. Figure 2 is the chip layout and Fig. 3 is a photograph of the monolithic gradient SQUID. The two junction SQUID has its inductance split into two parallel loops (L_S) . It is insensitive to a uniform field perpendicular to the loops since currents induced in the two SQUID inductances cancel each other at the junctions. However, it responds to a difference in field in the loops (dHz/dx). The SQUID inductances are very small, so to increase the field sensitivity, superconducting dc transformers are used to couple flux from the input pick-up loops (L_D) to the SQUID. The magnetometer fits on a one centimeter square silicon chip.

Each pick-up loop is 9mm by 3mm, which gives an inductance of 24 nH. To maximize the flux coupled to the SQUID, the pickup loop inductance and the primary inductance of the flux transformer, (Li) should be equal. In that case, half of the coupled flux is transferred to the SQUID. To accomplish this and strongly couple the input inductance to the SQUID inductance, a 21 turn coil flux transformer was used. This inductor is made of 5 um wide microstrip tightly coupled to the SQUID with a calculated L of 24 nH. The SQUID inductor is formed by a Nb thin film with a 5 um wide slot with a square 30um x 30um hole at each end. Each hole has an inductance of 50 ph, giving a total SQUID inductance of 25 pH plus the parasitic inductance of the slot. To reduce the parasitics, a superconducting flap, insulated on one side, covers the slot. However the input coil tightly coupled to the SQUID reduces the SQUID inductance further. Assuming a coupling constant k = 0.8, we calculate the SQUID inductance at 15 pH. The critical current was designed for about 40 uA with a beta less than 2, and the junctions were damped with l ohm resisters to eliminate hysteresis in the I-V characteris-All current and voltage leads are brought in symmetrically on the top and bottom of the SQUID. The gate current is fed in on coplanar type lines and external flux bias leads to the pick-up loops are microstrip lines to decrease magnetic coupling of bias currents to the SQUID.

2.2 SOUID Fabrication

The first thin-film SQUIDs were made using Nb base-electrode junctions with a lead alloy counter-electrode. The following six layer process was used:

- 1. First, a Nb film was patterned to form the SQUID inductor, base electrode, pick-up loops, current bias and voltage leads.
- 2. SiO was deposited to define the junctions and insulate part of the SQUID.
 - 3. Resistors were deposited to shunt the junctions.
- 4. The exposed Nb was oxidized to form the junctions, and Pb alloy counter-electrodes were deposited. This film also formed flaps over the slot in the SQUID inductance to reduce the



- 5. SiO was deposited to insulate the input coil from the SQUID.
- 6. Nb was deposited for the input coil and wiring interconnections.

After a number of unsuccessful attempts and considerable effort to operate the Joule-Thompson valve, we decided to make an all-niobium SQUID and mount it on the final stage of the Stirling refrigerator and operate around 7 K. The all Nb circuit technology was not initially available, but was developed during the contract with other TRW funds. To switch to an all Nb circuit, we changed two masks and used a tri-layer selective etching process to fabricate the Nb circuits. A Nb-Al₂O₃-Nb tri-layer junction covered the whole wafer and was patterned as in #l above. Then junctions were defined by reactive plasma etching. Next a single SiO layer was deposited with vias to contact the junctions. The circuit was then completed with the resistor and the Nb wiring layers.

2.3 SQUID Performance

A representative I-V curves with different applied fields is shown in Fig. 4(a), and the corresponding voltage modulation with fixed gate current in Fig. 4(b). The field current was applied to the center of one of the input transformers. Assuming that the current divides equally between the pick-up loop L_p and the input inductor L_i , the mutual inductance of the input coil and the SQUID is 0.5 nH. If the current is applied directly to the SQUID, the SQUID film inductance obtained from the flux period is 10 pH. This implies a coupling constant k equal to 0.7. The all Nb SQUIDs which didn't have the flap to reduce the parasitic inductance had an inductance nearly twice as large.

2.4 Magnetometer Cryocooler Interface

When the magnetometer was cooled in the Stirling cycle refrigerator, the SQUID silicon chip was mounted on a printed circuit board as shown in Fig. 5. This board was also made of silicon for good heat transfer to the final stage of the refrigerator. The board was mechanically clamped with heat-sink compound to a copper block threaded into the final stage of the Stirling cycle refrigerator as shown in Fig. 6. Electrical leads were attached to the chip by wire bonding and to the board by soldering. Low thermal conductivity manganin wire connected the board to room temperature. With an all Nb magnetometer installed in the cryocooler, we were able to reach the Nb transition temperature as evidenced by a zero voltage critical current of the SQUID. However the temperature was not low enough to operate the device as a magnetometer, because of the reduced current densities of the junctions.

3. Cryocooler

TRW successfully installed and operated the five stage Stirling cycle helium refrigerator developed at The National Bureau of Standards (NBS). The cryocooler uses two pneumatically driven diaphragm compressors which operate on the He working gas. These compressors, the pressure wave generator (PWG) and the displacer drive (DD) are controlled by solenoid valves which determine the timing and duration of their strokes. In addition, there are several manual metering valves in both the He and compressed air lines to control the amplitude of the pressure wave and the displacement.

We chose a Commodore 64 computer as an inexpensive yet very versatile controller to operate the cryocooler. The binary output of the computer operated the solenoid valves, and established the frequency of operation and the timing of the phases in the cycle. Any timing value could easily be changed by setting a byte in the memory. We also added an analog-to-digital board to monitor and record the temperature. This was used to try to optimize the refrigerator operation. Pressure transducers were installed in the PWG and DD lines as additional diagnostic support to the operation of the cryocooler. These waveforms could be recorded on an oscilloscope and compared with traces taken at other times.

We operated the cryocooler continuously for over three weeks on several occasions and achieved a minimum temperature below 7 kelvin. However, the operating temperature was neither reproducible nor extremely stable. The cryocooler required frequent re-adjustment of the manual needle valves to obtain optimum operation.

Figure 7 is the schematic diagram of the cryocooler. The PWG is controlled by the solenoid valves V1, V2 and V5. When V1 opens, compressed air expands the diaphragm and the helium gas in the refrigerator is compressed. The gas is expanded by opening the exhaust valves V3 and V5, relieving the pressure on the diaphragm. The movement of the displacer, which shuttles the gas between the warm and cold ends of the refrigerator at constant pressure, is actuated by the displacer drive and controlled by solenoid valves V2 and V4. Opening V2 drives the displacer piston up, and opening the exhaust valve V4 allows the displacer to move down.

The manual valves V_{m1} through V_{m5} are for fine tuning the system. Valve V_{m1} is a small leak to ensure equal average pressure in the refrigerator and the displacer drive. The gases on these two sides are isolated. The "working" gas of the refrigerator is in the pressure wave generator. Valves V_{m2} and V_{m3} to control the stroke of the displacer. Valve V_{m4} is used to maintain a constant average pressure in the cryocooler which can decrease due to leaks or cooling.

The seven phases of the valve sequences for one cycle of the Stirling cycle are described below. (Refer to Fig. 7 for the solenoid valve numbers). Figure 8 is a schematic diagram of the pressure in the refrigerator over a cycle.

- l. When the displacer has nearly reached the bottom and the sinking gas is at the warm end of the cryocooler Vl opens, driving the diaphragm in the PWG and compressing the He gas in the refrigerator.
- 2. Exhaust valve V4 of the displacer drive closes, ending the downward movement of the displacer.
- 3. V1 closes, ending the compression, and V2 opens, moving the displacer up and transferring the compressed gas to the cold end.
- 4. V2 closes, ending the movement of the displacer and exhaust valve V3 opens, decreasing the pressure from the PWG, and the gas is expanded.
- 5. A second PWG exhaust valve V5, which is in parallel with V3 which opened in phase 4, also opens to ensure complete expansion.
- 6. Exhaust valve V4 of the DD opens, allowing the displacer to fall downward and transfer the expanded gas to the warm end.
- 7. Exhaust valves V3 and V5 of the PWG close in preparation for the next compression.

This cycle is then repeated.

3.1 Controller Operation

A Commodore 64 computer was used as the controller to open and close the compressed air solenoid valves which move the diaphragm on the displacer and pressure wave generator drives. All sequencing and timing was done by software. Five of the eight output bits from the User Port are used to drive 5V relays after being conditioned by a TTL hex inverting buffer. relays operate the 110V ac solenoid valves. The main program to run the refrigerator is written in machine language and uses interrupts to gain control of the computer as shown in Fig. 9. By doing this, other programs may be run on the computer to optimize, read temperature, display valve status, etc., while the refrigerator timing control program is being run in the back-The program uses one of the internal timers to generate a non-maskable interrupt about every millisecond. When this happens the program takes control and checks if it is necessary to change the valves. A cycle consists of 5 phases of valves settings and corresponding times. The computer checks the current phase at 1 msec intervals to see if it is finished. it isn't complete, control is returned. If it has timed out, the valve settings and time for the next phase are read from a table in memory and the new valve bits are sent to the output port

before returning control. The timing or valves for any phase can be changed by writing new bytes in the valve and timing tables (i.e. in BASIC POKE table, byte). The overall period of a cycle can be changed by changing how often an interrupt is generated. This is done by writing two bytes to the internal timer.

Programs written in BASIC can be run concurrently with the machine language program and perform tasks such as reading and displaying temperature, and optimizing the cooler by changing the valve timing.

Using a program written in BASIC, we have attempted to optimize the timing for these phases. The program reads the temperature of the coldest stage (carbon resistor), changes the timing of one phase at a time, and reads the change in temperature after about 30 to 60 minutes. When this has been done for all phases, the timing change which has made the best improvement becomes the new time for that phase and the process repeats. The difficulty in doing this is that the response time is very long and one iteration can take 10 to 20 hours. During this time the operation of the refrigerator often changes and fine tuning of the manual valves is needed. The best values found for the valve timing phases are:

- 1) 120 ms
- 2) 280 ms
- 3) 230 ms
- 4) 480 ms
- 5) 130 ms
- 6) 60 ms
- 7) 280 ms.

Using these parameters, a typical optimized temperature of the final stage was 7 K. The first four stages had nominal temperatures of 175, 75, 30 and 16 K. The refrigerator would operate continuously for about 2 to 3 weeks with _me manual adjustments.

3.2 Cryocooler Modifications

Before operating the cooler, it was necessary to replace several defective valves in the original assembly with bellows sealed valves, and to machine two of the displacer stages and the displacer piston. The plastic displacer parts did not have sufficient clearance to operate smoothly when the cooler was assembled at TRW. This was evidently caused by swelling in the moist air when the system was disassembled. We subsequently found it helpful to out-gas the plastic parts under vacuum overnight before assembly whenever the system was disassembled for long periods of time.

As in all refrigerators, moisture is a major problem. Another problem is the diffusion of water molecules through the neoprene diaphragms in the PWG and DD. To minimize this source of moisture in the cooler, we installed an air drier on the compressed air input and also pumped on the space between the

double layered diaphragms.

NBS added a Joule-Thompson (J-T) stage to the final stage in order reach 4 K and operate Pb-alloy based Josephson devices. We were unable to operate the J-T valve satisfactorily. Because of the low flow rates required and thus the very small orifice, the valve clogged and could not be cleared. We fabricated several replacement throttling valves with no success in achieving the desired flow rates. A picture of one of the fabricated J-T stages is shown in Fig. 10. We decided to remove the J-T from the cryocooler and switch from the Nb-Pb to an all-Nb process for SQUID materials.

Under this program we also designed and fabricated new components for the pressure wave generator and the displacer drive. These components were designed in consultation with Dr. J. Zimmerman at NBS, who designed and built the cryocooler. also designed and constructed an entirely new self-contained compressor/gas handling system. The gas handling valve and piping schematic is shown in Fig. 11. The system is housed in a chassis occupying about 4 cu ft as shown in the photograph in Small cylinders for the compressed air ballast, high pressure He storage, low pressure He make-up reservoir, and He filter are housed in the bottom section of the unit. For easy access, the bellows sealed metering and shut off valves, regulators and pressure guages are mounted on the front panel. only connections necessary for operation are dry compressed air, He for filling the storage cylinder, and vacuum for initially purging the system. These connections are located on the rear panel. Under Zimmerman's direction, we also fabricated the fiberglass-epoxy sleeves for another cryocooler assembly. Using a lathe, Kevlar cord was wound one layer at time on a mandrel coated with a mold release. After each layer, epoxy resin was brushed on and allowed to set slightly before continuing with the next layers.

3.3 Refrigerator Operational Procedures

PSYSTEM RESISSION PROFITE PSYSTEM

- 1.) Purge: After He lines from the displacer drive and pressure wave generator are connected to the system, purge by pumping and flushing with ultra pure Helium gas numerous times (>10). Be sure all He line valves are fully open.
 - 2.) Charge system with pure He gas to 40 psi.
- 3.) Turn on dry compressed air (80 psi) to displacer and PWG drives.
- 4.) Turn on controller (Commmodore-64) to the solenoid air valves. These are 5 volt signals.
- 5.) Close the needle valve between the displacer drive and the pressure wave generator drive (V_{m1}) , and then open 1/2 turn. There should be practically no flow through this valve, it is only to assure equal average He pressure between the displacer and pressure wave generator drives.

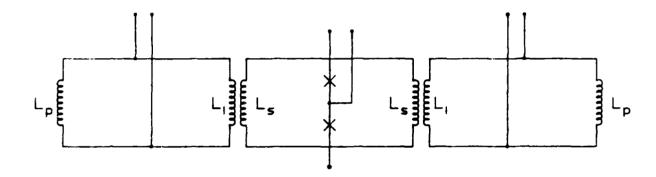
- system until no pressure fluctuation of gauge. Again there should be no five terms replenishes any He that might lear of the state of t
- 7.) Adjust the valve from the ingred displacer (V_{m2}) for smooth operation is system is warm, the motion is often in as it cools. When the system is turned down to prevent the displace is bottom of the stroke. The final temperature the setting of this valve.
- 8.) The cooling of the refrigerat measuring first the upper stage there ones as they begin to cool. It takes the cool, at which time the carbon resistant the final stage can be monitored. The strict reduced by adjusting the manual value of the solenoid valve (V_{m3}) on the displace.

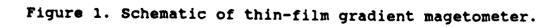
4. Conclusion.

TRW successfully transferred the territory operate, and fabricate the small split-line. from NBS to industry. Performance was fine + which was demonstrated at NBS. We also desired successfully tested a planar thin file rate to the niobium technology. A difficulty in operation was the frequent requirement to manually adjust optimum performance. For future use, we recomme monitor operating parameters such as the dynamic prodisplacer position under computer control and aut mai these adjustments. The Joule-Thomson stage als: 7 feasible because of the difficulty of making and raceprecision small orifice. We would like to acknow. 6:19 Zimmerman for his assistance in the design of new and the operation of the cryocooler, and M. K. Wagner to be fabrication of these components.

Figures

- 1. Schematic of thin-film gradient magetometer.
- 2. Pen plot of the planar SQUID and input coils.
- 3. Photo of the magnetometer chip.
- 4.(a.) Flux modulation of the current-voltage curves of the SQUID.
 - (b.) Voltage modulation vs. applied field.
- 5. Photo of silicon pc board with chip attached.
- 6. Sketch of the attachment of the pc board to the lower stage of the cryocooler.
- 7. Schematic of cryocooler.
- 8. Timing diagram of the cryocooler cycle.
- 9. Assembly language listing of the control program.
- 10. Photo of original (left) and new Joule-Thompson stages.
- 11. Schematic of new gas handling valve and piping.
- 12. Photo of compressor and valving assembly.





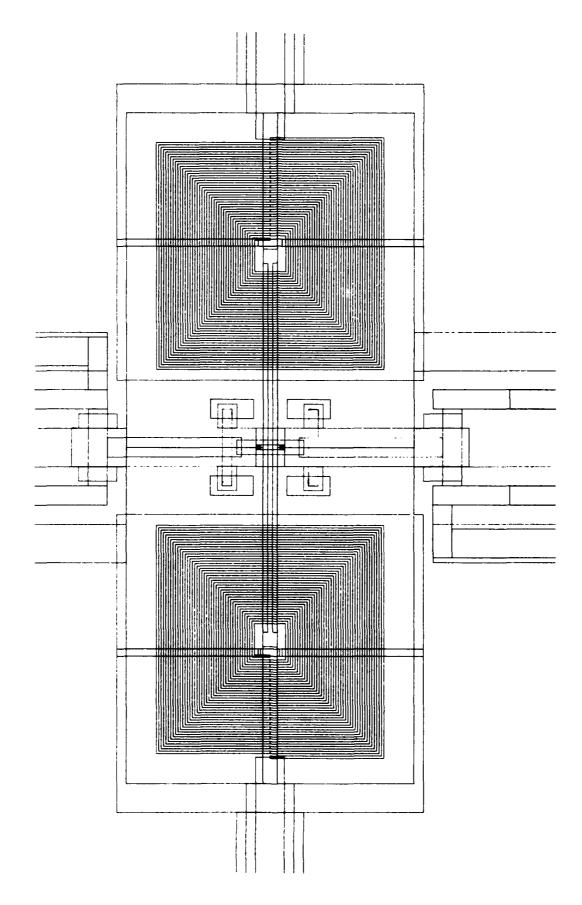


Figure 2. Pen plot of the planar SQUID and input coils.

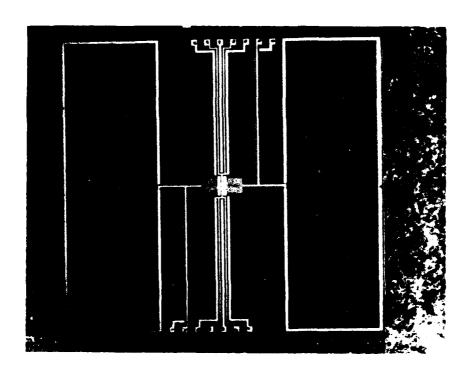
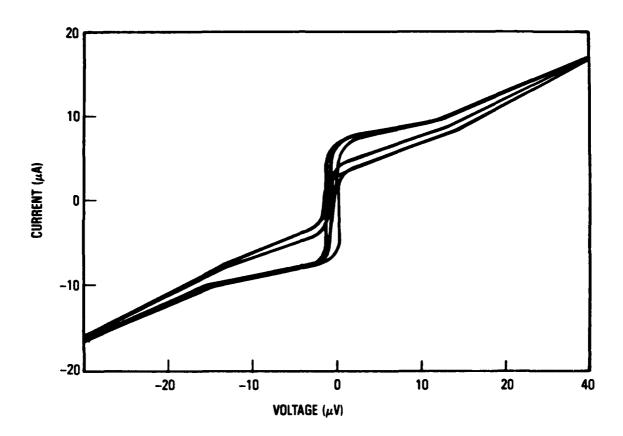


Figure 3. Photo of the magnetometer chip.







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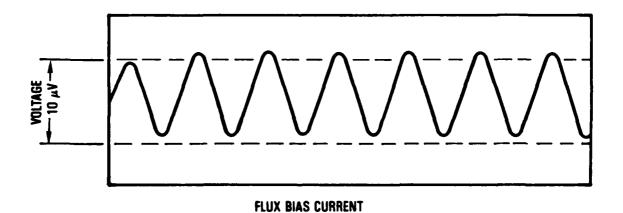
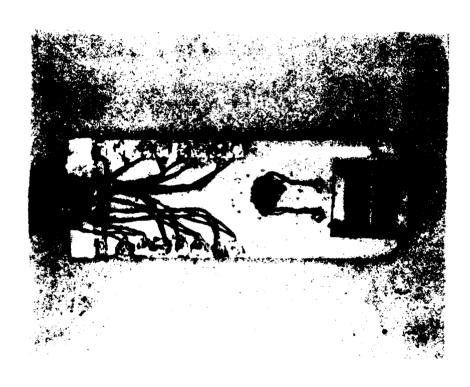


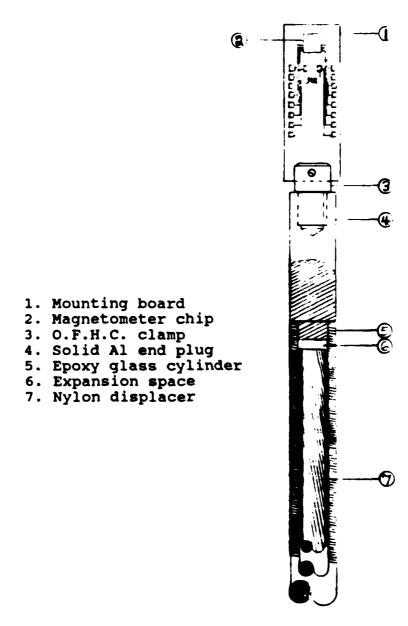
Figure 4.(a.) Flux modulation of the current-voltage curves of the SQUID. (b.) Voltage modulation vs. applied field.



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Figure 5. Photo of silicon pc board with chip attached.



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Figure 6. Sketch of the attachment of the pc board to the lower stage of the cryocooler.

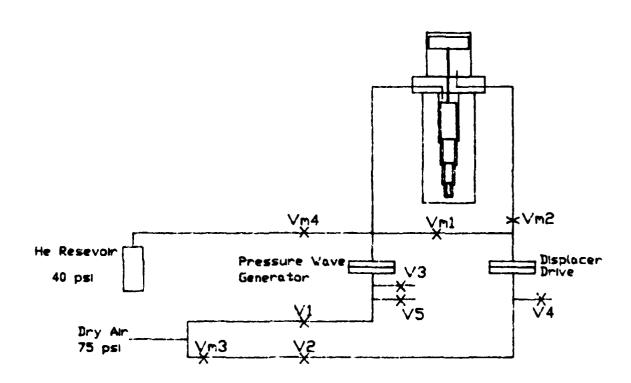
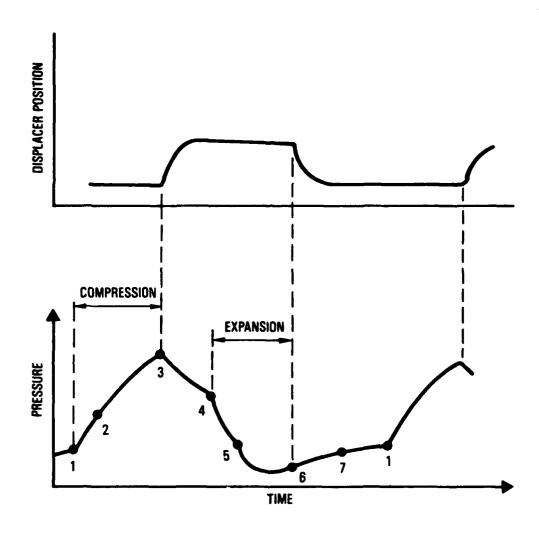


Figure 7. Schematic of cryocooler.



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- 1. VI OPENS 2. V4 CLOSES
- 3. V2 OPENS, V1 CLOSES 4. V3 OPENS, V2 CLOSES
- 5. V5 OPENS
- 6. V4 OPENS 7. V3, V5 CLOSE

Figure 8. Timing diagram of the cryocooler cycle.

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95	49328 173 201 1	* 2	LDA DF					
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97	49334 149 1		LDA #1			1 MMITTE	CHAR	
48 48	49776 157 71 2 49779 7H2		STA CO	COP.				
11111	4974 208 234		BNF LF					
101	49 1. 76		RTS					
100	PV 1 1	1.1 ***	EVE 10					
t * · · *	40-14	\$ \$4 _{6.5} ° 1	4.1					
104	47345	CNUM	841 S	09				
•								
105	49346	1RC	D Y7 •	00				
0								
0 0	4 9347 0 0	TEMP	941 8	00000000	,			
107	49351	ON	841 B					
86 0				-				
108	49353	OFF	BYT B	7				
6 7 0								
109	49355	MEM	BYT .	00				
110	51200		EQU S	CBOO				
111	51200	VTBL	BYT &		14140402	20201		
• •	8 20 20 20	4 2	2 1					
112	51210	TIBL	BYT :	IEFA3CO	ENFAEA16	FA7B		
	0 60 130 230 25	230 30	250 120					
	51220	TIMER	H BYT 8	E003 1	-1000M5.			
113								
232 3	i=0				1802	56589	~~~	
232 3 ERRORS	792 TLDM	56587	CRB	54591				•2042
232 3 ERRORS NHIV SCREEN	792 TLDM	54582 55727	CRB VOUT	56571 56577	BORDER	51280	ne G	49152
232 3 ERRORS NRIV SCREEN SETUF	792 TLDM	55727 49208	VOUT START	56577 49232	BORDER	57290 4924A	ne G	49152
232 3 ERRORS MMIV SCREEN SETUF EXIT	792 TLDM 1 1055 COLOR 49152 \$10F 49284 CONTU	55727 49208 49294	VOUT START VIEW	56577 49232 49308	BORDER	57290 4924A	ne G	49152 49262 49731
232 3 ERRORS NMIV SCREEN SETUF EXIT TIME	792 TLDM 1 1055 COLOR 49152 STOF 49284 CONTU 49343 PHASE	55727 49218 49294 49344	VOUT START VIEWV CNUM	54577 49232 49308 49345	BORDER	57290 4924A	OPG STEE NX TEMP	49152 49262 49331 49347
232 3 ERRORS NHIV SCREEN SETUF EXIT TIME ON	792 TLDM 1 1055 COLOR 49152 \$10F 49284 CONTU	55727 49208 49294	VOUT START VIEWV CNUM	56577 49232 49308	BORDER	57290 4924A	ne G	49152 49262 49731



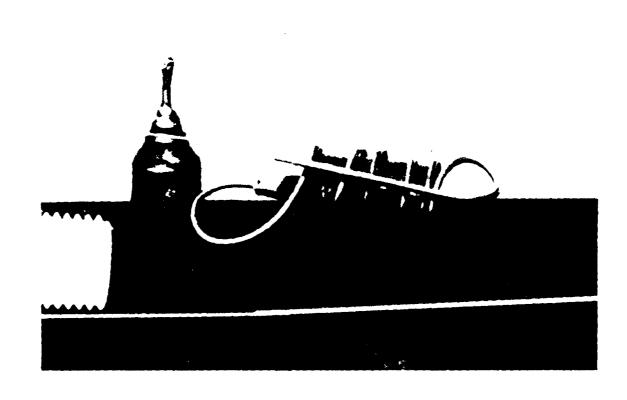
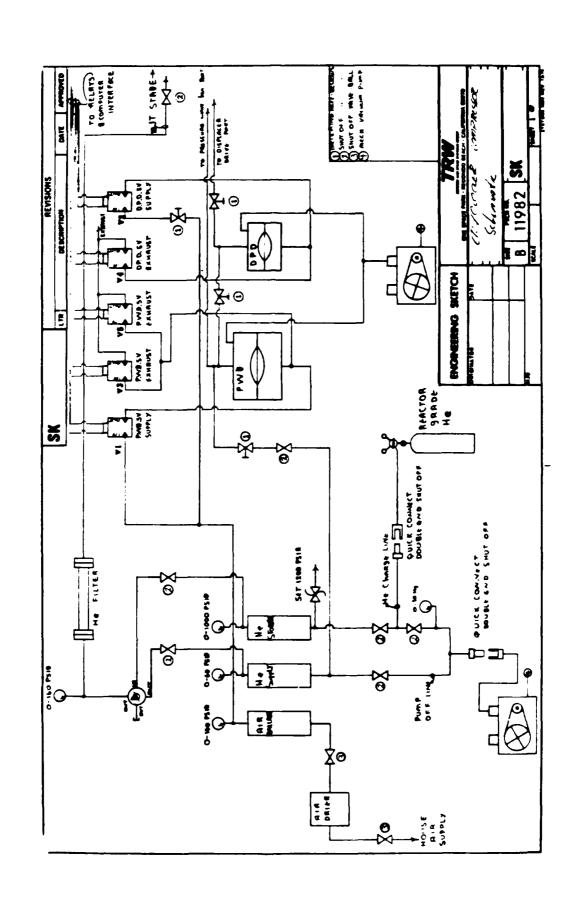


Figure 10. Photo of original (left) and new Joule-Thompson stages.



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Figure 11. Schematic of new gas handling valve and piping.

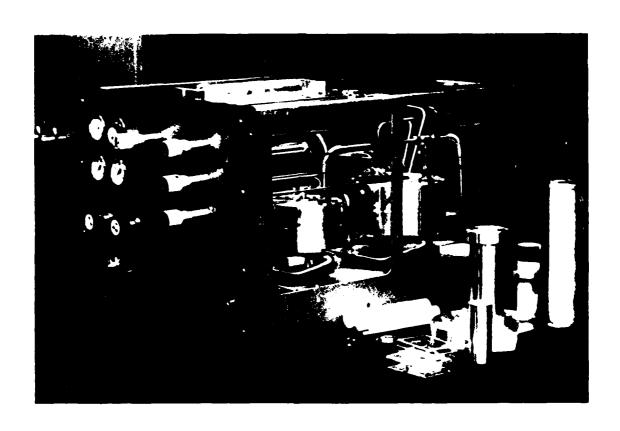


Figure 12. Photo of compressor and valving assembly.

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